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CORROSION CRACKING AND HYDROGEN EMBRITTLEMENT OF THERMALLY HARDENED REINFORCEMENT OF PRE-STRESSED REINFORCED-CONCRETE STRUCTURES

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13. ABSTRACT

Steel samples with the compns. C 0.15-0.25, Mn 0.25-1.5, Si 0.67-1.36% and also some alloys having V 0.12, Al 0.1 and Ti 0.11% have been studied during thermal-mech. hardening. Testing time ranged 2.5-21 hr. Steel tempered at temps. lower than annealing had an increased tendency to corrosion cracking. Even at high temps. (470-600°) it did not acquire satisfactory corrosion stability. All specimens of the thermally hardened steels broke down in brittle pieces without the formation of any visual corrosion products in 40-60 min. when subjected to a H_2S satd. aq. soln. at room temp. High stable cold worked steel specimens were unaffected even after 200 hr in the above soln. The disintegration is due to H embrittlement as shown by cathodic polarization curves. The same specimens were kept in a hermatically sealed chamber in HCl vapor (2 mg/l.) at 15-17°. All samples cracked within 20 hr. Cold worked sample showed intensive general corrosion but cracking was not seen. Effect of cathodic polarization on the time required for cracking has also been studied. Stress corrosion cracking in 10% NH_4CNS soln. was also due to H embrittlement. Cathodic protection, action of stray currents, etc., can also give rise to H embrittlement resulting in the stress corrosion cracking of the above structures. [AT0047047]

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Titanium						
Tantalum						
Niobium						
Aluminum						
Boron						
Calcium						
Ammonium Nitrate						
Hydrogen Sulfide						

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* ye initially, after vowels, and after ъ, ь; e elsewhere. When written as ё in Russian, transliterate as yё or ё. The use of diacritical marks is preferred, but such marks may be omitted when expediency dictates.

CORROSION CRACKING AND HYDROGEN EMBRITTLEMENT OF THERMALLY HARDENED REINFORCEMENT OF PRESTRESSED REINFORCED-CONCRETE STRUCTURES

Candidates of Technical Sciences S. N. Alekseyev, G. M. Krasovskaya and Engineer E. A. Gurevich

In the corrosion of high-strength reinforcement of prestressed reinforced-concrete structures, their destruction for the most part comes about suddenly. This is explained by the following. Prestressed reinforcement as compared with ordinary reinforcement has little deformation and absorbs high tension. The already small decrease in section in the place of corrosion damage gives rise to excess of ultimate strength of the remaining section of reinforcement. Furthermore, such specific kinds of corrosion as corrosion cracking and hydrogen embrittlement are inherent to reinforced steels.

"Stress corrosion of metal" usually means the processes which are developed while the metal is being acted upon by tensile stresses and agressive medium. The damages which appear as a result of stress corrosion differ sharply in appearance from the damages which are developed with ordinary electrochemical corrosion. They are characterized by formation in the metal of fissures without significant surface corrosion damages.

Depending on the nature and conditions of the effect of agressive medium, the brittle rupture of metal under stress can occur either as a result of corrosion cracking, or due to hydrogen embrittlement.

Corrosion cracking of metals is most often caused by media in which the processes of anodic dissolution are strongly localized (usually in the absence of a noticeable general surface of corrosion). The strength of localized corrosion can be quite significant, as a result of which the development of very narrow depressions progresses. These local corrosion damages together with all possible defects which are formed on the metal surface after heat treatment and machining are the primary stress raisers.

The anodic process of dissolving of metal on the bottom of the stress raisers goes easier than in surface sections, because of increased stresses which promote destruction of the protective film. During the second period of corrosion cracking the primary stress raisers develop into microcracks whose depression is accompanied by the accelerated deformation of metal and by the appearance of new voltaic couples having an anode section on bottom and cathode sections on the walls of fissures. At this point of cracking, the rate of process will be affected most by the conditions of corrosion in fissures.

Corrosion cracks are narrow slotted channels filled with corrosion products. The access of oxidizer to their bottom has been made difficult as compared with surface sections. The anodic process on the bottom of fissures goes easier since restoration of protective film is sharply retarded due to the fact that access of oxygen has been made difficult. In the third period of development of corrosion cracking the fissure rapidly increases, and with decrease in cross section of metal to critical value,

final brittle rupture comes about. In the period of "avalanche" destruction the dominant role is played by the mechanical factor; electrochemical factors during this period have very little effect.

During hydrogen embrittlement of high-strength steel, as with corrosion cracking, tears are formed, and brittle breaks of stressed elements occur. However, the mechanism for destruction in this case is another. The destruction of the stressed metal as a result of hydrogen embrittlement is usually caused by absorption of atomic hydrogen. As a result of the corrosion process hydrogen is liberated in cathode sections in this sequence: first occurs discharge of hydrated hydrion (in acid media) or ionization of water molecules (in alkaline media) with formation of an adsorbed hydrogen atom, and then comes recombination of hydrogen atoms.

Atomic hydrogen usually being formed is recombined on the metal surface into molecular hydrogen in a short time, but sometimes this process is slowed down by so-called catalyst poisons [1]. One of the known catalyst poisons is hydrogen sulfide, which retards the second stage of cathode reaction and simultaneously accelerates the anode reaction of metal solution.

Acceleration of the anodic process gives rise to an increase in the rate of formation and concentration of atomic hydrogen on the cathode, which gives rise to its diffusion and creation of pressure in the pores of metal. When external tensile stresses and localized corrosion destruction exist, along which a crack develops, pressure which develops in microspaces will promote rapid development of the crack and lead to the brittle rupture of metal.

Numerous investigations [2, 3] of corrosion resistance of structural metals and alloys show that the inclination of metal toward stress corrosion depends basically upon its structure

which in turn is determined by chemical analysis and by the conditions of heat treatment and machining.

Taking into account the wide application of thermally hardened reinforcement in the production of prestressed reinforced-concrete structures, it is necessary to study the effect of the chemical anslysis of steel on the inclination toward corrosion cracking, of the mode of heat treatment on the stability of high-strength reinforcement during stress corrosion, and of the basic industrial media on the ability to cause brittle rupture of stressed thermally hardened reinforcement.

The influence of alloying on the corrosion resistance of thermally hardened reinforced steel in literature has not been fully enough illuminated. It is known that thermally hardened steel with high corrosion resistance has been obtained in the Federal Republic of Germany. F. Dyuma [4] reports that thermally hardened steel, practically not inclined toward cracking has been obtained by decreasing (halving) the content of carbon and manganese while simultaneously raising (tripling) the content of silicon and adding chromium in an amount approximately equal to the carbon content.

F. F. Azhogin [5] showed that with the raising of carbon content from 0.3 to 0.78% the inclination of thermally hardened steel toward corrosion cracking increases.

However, it is known that thermally hardened reinforcement of marks St. 5 and 35GS steel with carbon content 0.29-0.35% has minimum resistance to cracking while high-strength hard-drawn steel wire with carbon content 0.79-0.8% does not lean toward stress corrosion [6]. This is evidence of the fact that only one change in the carbor content of steel can substantially affect its inclination toward corrosion cracking.

According to F. F. Azhogin [5], corrosion resistance is adversely affected by an increase in the chromium, manganese, and nickel content of steel. A number of authors believe that the more nickel a steel contains, the more resistant it is to cracking. In all cases with increase in tempering temperature, the inclination of steel toward cracking drops.

An increase in silicon content to 1.78%, on the contrary, can increase the corrosion resistance of steel with tempering above 350°C.

There are data [3, 7] about the fact that the inclination of steel toward corrosion cracking can be decreased by means of alloying with titanium, tantalum, niobium, aluminum, and boron to the extent T = 4C, Ta = 16C, Nb = 8C (C is the carbon content in the alloy), Al = 0.5%, B ≐ 0.005%.

The most widespread method of development of the inclination of various forms reinforced steels toward brittle rupture is the test of the stressed samples of reinforcement in boiling solutions of calcium and ammonium nitrate.

In the central laboratory of corrosion accelerated tests were conducted in a solution of composition 600 parts by weight $Ca(NO_3)_2$; 50 parts by weight NH_4NO_3 and 350 parts by weight of water.

Tensile stresses during test can be created, maintaining either constant deformation, or load. In both cases this can be achieved both by central elongation of sample and by curvature.

The basic component of samples was tested with tension of reinforcement to rigid frames.

Comparative accelerated tests of reinforcement of small diameter (up to 6 mm) were conducted with the samples, stresses in which were created by means of curvature of samples into a bracket around the mandrel of the appropriate diameter or within the frames.

The value of the stresses being controlled of thermally hardened reinforcement during accelerated corrosion tests was taken equal to ~ $0.75\sigma_{B}$.

Today we together with the experimental design office of TsNIISK [Central Scientific Research Institute of Structural Parts] im. Kucherenko have developed a lever installation for testing bent reinforced samples for inclination toward corrosion cracking with constant stress on the boundary fiber of the sample.

During the conducting of tests for recording the break of a rod we used a timer whose construction has been developed together with the laboratory of measureing equipment NIIZhB [Scientific Research Institute of Concrete and Reinforced Concrete] (Fig. 1).

The instrument has a weekly clockwork. Anchored to the cylinder being rotated is film with hourly divisions, over which the pens of the automatic recorders glide. The number of automatic recorders corresponds to the number of samples being simultaneously tested. When the rod breaks the current circuit is interrupted, the pen of the automatic recorder moves away from the cylinder, and writing stops.

We tested[1] industrial batches of thermomechanically hardened reinforcement of the Krivoy rog Metallurgical Plant for inclination

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[1]E. M. Filippov and V. N. Frolov took part in the work.

Fig. 1. The automatic recorder which records the break of rod.

toward corrosion cracking (Table 1). Melts were distinguished by content of carbon (0.15-0.25%), manganese (0.25-1.5%), and silicon (0.67-1.36%), and part of the melts was alloyed with vanadium (0.12%), aluminum (0.1%), and titanium (0.11%).

It is hard to establish the influence of individual components on corrosion behavior of steel from these tests because different melts were used.

The tendency of steel toward stress corrosion was judged from time to cracking, which varied for various melts from 2.5 to 21 hours.

Thermally hardened reinforcement tested showed much inclination toward corrosion cracking. Therefore, it is expedient to limit its use in constructions which work in aggressive media.

It is known that the mode of heat treatment and especially the tempering temperature substantially influence the corrosion

Table 1. Chemical composition, speed-torque characteristics and time to cracking of thermomechanically hardened reinforcement of the Drivoy rog Metallurgical Plant.

Diameter in mm	Chemical composition in. %								σ_B, kgf/mm^2	$\sigma_{0.2}$, kgf/mm^2	σ_s, kgf/mm^2	Time to cracking in h
	C	Mn	Si	S	P	V	Al	Ti				
10	0,2	0,93	0,99	0,023	0,011	0,12	—	—	118—132	100—105	95—102	2,5—7,5
10	0,19	1,02	0,67	0,027	0,012	—	—	—	113—136	101—110	89—98	2,5—4
10	0,18	1,42	1,06	0,035	0,011	—	—	—	123	103	92	5
10	0,2	1,5	1	0,033	0,016	—	—	—	125	105	93	5—7
10	0,18	1,15	0,93	0,034	0,015	—	0,1	—	125—133	99—106	93—99	10,5—13,5
10	0,2	1,31	1,36	0,025	0,014	—	—	—	130—137	105—108	89—103	15—17
12	0,15	1,12	0,72	0,045	0,017	—	—	—	112—116	91	83—86	6—10
10	0,17	1,05	0,92	0,026	0,025	—	—	0,11	126	98	[illegible]	7—9,5
10	0,16	0,86	0,9	0,021	0,012	0,06	—	—	120—136	103—105	90—102	13—21
10	0,25	0,25	0,88	0,032	0,012	—	—	—	136—145	105—110	102—108	12—13
10	0,2	1,17	0,9	0,016	0,031	—	—	—	119—129	103—109	90	6

behavior of steel: with raising of tempering temperature the tendency toward cracking is lowered. At the Makeyev Metallurgical Plant an experimental batch of reinforcement was released (with diameter of 14 m ` which was thermally hardened from rolling heating. The reinforcement was made of 35GS steel of one melt (0.33% C; 1.08% Mn, 0.72% Si; 0.29% S, 0.016% P). After strengthening the strength of classes At-IV; At-VI; At-VII was obtained. The tempering was accomplished spontaneously because of the heating by hot internal layers of external layers which were cooled during hardening. The tempering temperature in this case was regulated by the duration of holding in water (Table 2).

Accelerated corrosion tests showed that at greater strength lower tempering temperature) steel had an increased inclination toward corrosion cracking: At-VII - 3 h, At-VI - 5 h, At-IV - 20 h. Even at high tempering temperature (470°C) thermally hardened 35GS steel obtained did not have satisfactory corrosion properties.

There are data about the fact that steels tempered at 600°C, have not been subjected to corrosion cracking. But with such tempering the strength characteristics of steel are sharply lowered. Obviously research showed continue on methods and modes of thermal hardening and selection of the proper chemical composition of reinforced steel, support, and corrosion ratio. One of the variants of treatment of carbon steels for raising their strength characteristics and corrosion resistance is additional tempering of volumetrically hardened metal [8]. The use of such heat treatment allows obtaining articles with high-strength center and comparatively soft surface layer. Destruction under the prolonged effect of stresses and aggressive medium in most cases begins from the surface. Increase in the plasticity of the surface layer should raise cracking resistance. Apparently, this method is a promising one that allows obtaining thermally hardened reinforcement resistant to corrosion.

Table 2. Speed-torque characteristics, mode of heat treatment and time to cracking of thermally hardened reinforcement of the Makeyev Metallurgical Plant.

$\sigma_{0.2}$, kgf/mm²	$\sigma_{в}$, kgf/mm²	δ_5, %	Время выдержки в воде в сек (1)	Отпуск (электроподогрев) до t в °C (2)	Время электроподогрева (3)	Класс арматуры (4)	Время до растрескивания в ч—мин (5)
82	100	15,2	2,2	440	—*	Ат-IV	12—20
86	98	15,7	2,43	470	3 min 45 s	Ат-IV	20
84	98	14,1	2,42	470	3 min 30 s	Ат-IV	22
109	125	10,7	2,63	380	—*	Ат-VI	2—5
117	133	10	2,8	380	—*	Ат-VI	4—7
118	134	10,2	2,85	390	2 min 17 s	Ат-VI	3,5—7
126	147	11	2,95	340	—*	Ат-VII	1,5—2,5
123	145	9,7	2,8	340	—*	Ат-VII	2
122	140	10,3	2,85	385	2 min 15 s	Ат-VII	3—5

KEY: (1) Holding time in water in s; (2) Tempering (electric heating) to t in °C; (3) Time of electric heating; (4) Class of reinforcement; (5) Time to cracking in h - min.

*Samples were not subjected to additional electroheating.

It has been established [9, 10], that the cracking of thermally hardened rod reinforcements made of St. 5 and 35GS, and also wire rod is possible under the effect of liquie media containing nitrates an chloride ions, in concrete with $Ca(NO_3)_2$ additive, in the case of anode polarization and in a number of other cases.

It was expedient to check some other industrial media for the ability to cause brittle rupture of thermally hardened reinforcement during stress corrosion as a result of both corrosion cracking and hydrogen embrittlement.

Hydrogen sulfide is a substance rather widely used in industry. It is liberated, for example, into the atmosphere of the spinning shops of the enterprises of man-made fiber, it is formed in

sewers, reservoirs, etc. The possibility is not preculded of the formation of hydrogen sulfide in the concrete prepared on cements having increased content of sulfides (aluminous slag and others).

Taking this into account stressed high-strength reinforced steels were tested during the action of hydrogen sulfide. Samples of high-strength hard-drawn wire 5 mm in diameter of class Vr-II and thermally hardened reinforcement 3 mm in diameter of class At-VI were investigated.

Stress of samples was created by their curvature into a bracket on frames. In this case effort in the extreme stretched fiber was 60-70% of ultimate tensile strength of the steel.

Tests were run in a saturated aqueous solution of hydrogen sulfide at room temperature. All samples of thermally hardened steel failed under these conditions in 40-50 min. The break of samples is brittle, without formation of visible corrosion products.

The samples of high-strength hard-drawn wire after a 200 h stay in a saturated aqueous solution of hydrogen sulfide did not have fissures. The effect of the medium was practically not reflected on the strength of the samples, but caused a certain decrease in residual uniform elongations and plasticity (number of bends).

To clarify the mechanism of brittle rupture of stressed thermally hardened reinforcement in a saturated aqueous solution of hydrogen sulfide the method of electrochemical polarization was used. The appearance or stop development of already generated corrosion cracks only when conditions of emergence of hydrogen embrittlement of steel do not exist. The influence of a polarizable

cathode current on the rate of cracking of thermally hardened wire in a saturated aqueous solution of hydrogen sulfide is presented in Fig. 2.

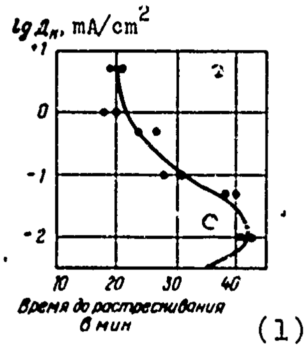


Fig. 2. Influence of cathode polarization on the rate of cracking of thermally hardened wire in a saturated aqueous solution of hydrogen sulfide. KEY: (1) Time up to cracking in min.

The pH value of the saturated aqueous solution of hydrogen sulfide is within the limits 4.2-4.5. The surface potential of stressed reinforcement at pH = 4.5 without superposition of cathode polarizable current is somewhat negative (-580 mV) than the potential of liberation of hydrogen (-450 mV). Under these conditions on the surface of steel a quantity of hydrogen is liberated which is sufficient for the development of the process of hydrogen embrittlement.

With increase in the density of cathode polarizable current, judging from the nature of the curve of cathode polarization (Fig. 3), the process of liberation of hydrogen in cathode sections is intensified, and although it flows with noticeable retardation, it gives rise to acceleration of cracking. Thus, without superposition of polarizable current and with cathode polarization, cracking of stressed thermally hardened reinforcement in a saturated aqueous solution of hydrogen sulfide occurs as a result of hydrogen embrittlement of steel.

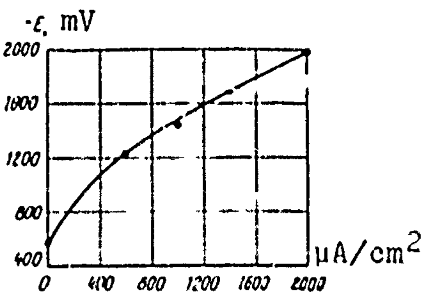
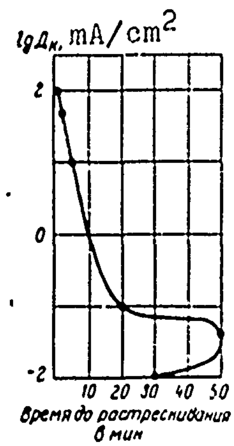


Fig. 3. Curve of cathode polarization of stressed thermally hardened wire in a saturated solution of hydrogen sulfide.

Probably brittle rupture of thermally hardened reinforcement connected with hydrogen absorption of steel is possible when it is acted upon by a majority of acid media. It has been experimentally established that reinforcement is cracked in hydrogen chloride vapors. Samples of thermally hardened reinforcement of class At-VI 3 mm in diameter and of high-strength hard-drawn wire of class Bp-II 5 mm in diameter whose stress was created with curvature and was about 70% of ultimate strength in an airtight chamber under the influence of hydrogen chloride vapors 2 mg/l in concentration at 15-17°C. All samples (6 pcs.) of thermally hardened reinforcement cracked in 20 h. High-strength hard-drawn wire which was under these conditions 1500 h, underwent intense general corrosion, but did not have fissures.

Analogous was the mechanism for brittle rupture of stressed thermally hardened reinforcement even during the action of ammonium NH_4CNS. Cathode polarization of stressed samples of thermally hardened wire 3 mm diameter in a 10% solution of ammonium rhodanide gave rise to pronounced shortening of the time to their destruction (Fig. 4).



(1)

Fig. 4. Influence of cathode polarization on rate of cracking of thermally hardened wire in 10% ammonium rhodanide solution at 50°C.
KEY: (1) Time to cracking in min.

Hydrogen embrittlement in ammonium rhodanide was uncovered also during research on thermally hardened reinforced steel St. 140/160 [11].

The phenomenon of brittle rupture of thermally hardened reinforcement as a result of hydrogen absorption should apparently be considered a type of stress corrosion. As experiments showed, the effect of a saturated aqueous solution of hydrogen sulfide on unstressed reinforcement does not give rise to its destruction. However, the prolonged stay of unstressed reinforcement in a hydrogen sulfide medium somewhat decreases residual relative and uniform elongations, and also plasticity both of thermally hardened wire rod and hard-drawn high-strength wire (Table 3).

It has been established that cathode or protector protection, and the action of stray currents causing cathode polarization can sometimes lead to the development of acid brittleness of the stressed thermally hardened reinforcement in concrete of constructions. In our experiments during action on stressed reinforcement of class At-VI 3 mm in diameter in dense concrete of constant potential of 1.2-1.5 V (on a calomel electrode) for 12-15 h brittle break of all samples came about without formation of visible corrosion products (Fig. 5).

Fig. 5. Form of destruction of thermally hardened reinforcement as a result of hydrogen absorption from the action of cathode polarizable current.

Conclusions

1. Thermally hardened reinforced steels develop a tendency toward corrosion cracking. In connection with this the given forms of reinforced steels can be used with complete confidence only in

Table 3. Change in speed-torque characteristics of high-strength reinforcement in saturated aqueous solution of hydrogen sulfide without stresses.

Reinforcement	Storage Condition	Breaking load in kgf	Residual elongation per unit length δ_{10}, %	Residual elongation per unit length δ_5, %	Residual uniform elongation δравн	Relative reduction of area ψ, %	Number of bends
Thermally hardened wire rod, the class Atk, 6, 5 mm in diameter	Air-dry	4830	7,25	10	4,35	20,8	3
	100 h in hydrogen sulfide solution	4880	6,45	9,5	3,7	20,3	2
High-strength hard-drawn wire of class Vr-II, 5 mm in diameter	Air-dry	3110	5,2	7,2	3,1	38,4	3
	100 h in hydrogen sulfide solution	3170	4,8	6,8	2,8	14,4	1

constructions intended for categories I and II of fissure resistance and being operated under nonaggressive conditions or weakly aggressive conditions in the absence of aggressive gases.

2. In a number of media destruction of stressed thermally hardened reinforcement can occur as a result of hydrogen embrittlement. Such a form of destruction in our experiments was obtained in a medium of hydrogen sulfide, hydrogen, chloride, and ammonium rhodanide.

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